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ROCKETDYNE

A DIVISION OF NORTH AMERICAN AVIATION, INC.

CANOGA PARK, CALIFORNIA

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COMPATIBILITY OF MATERIALS WITH HIGH TEMPERATURE POTASSIUM

SECOND QUARTERLY PROGRESS REPORT,

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FOREWORD

This second quarterly progress report is presented in fulfillment of requirements of National Aeronautics and Space Administration contract No. NAS-5-453.

ABSTRACT

This report, covering the period from 1 August to 31 October 1960, describes the second quarter of effort in a 13-month evaluation of refractory metals for potential use in high-temperature, turbo-electric, space powerplants using potassium as the working fluid. In isothermal-capsule tests at temperatures to 2000 F, corrosion of wrought columbium and columbium/1-percent zirconium alloy was negligible except when these metals were combined with nickel alloys. In 50 hours of Rankine-loop tests at temperatures to 1800 F, molybdenum resisted attack best of the refractory metals tested. Limited mechanical properties tests are also described. Two columbium alloy loops are under construction, and the first vacuum chamber is complete.

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INTRODUCTION

A 13-month research program to investigate the high-temperature compatibility of potassium with various refractory metals was initiated under contract No. NAS 5-453 by the National Aeronautics and Space Administration. It is being performed jointly by Rocketdyne and Atomics International, divisions of North American Aviation, Inc. under the management of Rocketdyne.

The major objective of the Rocketdyne program is the determination of materials compatibility with high-temperature potassium in Rankine-type loops. Three loops are to be operated with boiler temperatures of 1800 F, 2000 F, and 2200 F. The lowest temperature loop was achieved by modifying a boiling sodium loop built by Rocketdyne prior to the NASA contract. The remaining two loops are to be constructed of a columbium/1-percent zirconium alloy. The program includes supporting capsule studies which are being performed by Atomics International.

SUMMARY

Over 25 capsule experiments have been completed, and the experimental procedures have been perfected. The conclusions from these experiments are as follows:

1. When nickel and columbium alloys are placed in potassium at temperatures above 1450 F, a reaction layer forms on the columbium alloy, and a weight loss occurs.
2. Columbium alloys sealed in columbium alloy capsules show no appreciable attack.
3. Oxygen in potassium may promote nickel/columbium attack.
4. Columbium/1-percent zirconium alloys are not as readily attacked by nickel as is wrought columbium.
5. Welded regions of columbium alloys are particularly susceptible to intergranular attack by potassium.

The low-temperature potassium loop has been operated successfully for 50 hours at 1800 F boiler temperature. Samples of refractory metals were exposed to the potassium at the boiler outlet, and their resistance to corrosion was evaluated as follows:

Molybdenum	Most resistant
Columbium/1-percent zirconium	Second most resistant
Wrought columbium	Second least resistant
Tantalum	Least resistant

Two columbium samples, located downstream from the nozzle, were not found, and their fate is not known. Additional tests are in progress, and this part of the program will be completed soon.

Two high-temperature columbium loops have been designed, and the details are presented in the Technical Discussion. These are all columbium/1-percent zirconium loops employing electrodynamic pumps. The electrodynamic pump was selected because it appears to be capable of providing the required heads and flowrates in an all columbium system, and eliminates the danger of introducing contaminants that may confuse the compatibility studies. Welding studies, flow gage calibration tests, liquid level gage tests, thermocouple evaluation, and potassium purification procedure evaluations have been initiated to guide the construction and ensure successful operation of the loops.

A schedule of activities for the duration of the contract is presented. All parts of the loop have been ordered or fabrication initiated in the Rocketdyne shops; it is anticipated that the loops will be in operation near the end of January 1961.

TECHNICAL DISCUSSION

CAPSULE CORROSION STUDIES

The capsule corrosion studies are expected to provide basic information to assist the loop program. As discussed in the last quarterly progress report, the first capsules failed because of unsatisfactory capsule fabrication. These techniques have been perfected, and the proper equipment is in operation. The technique finally adopted is to arc weld a cap on a closed-end tube in a dry box containing purified argon gas. The gas is purified by passing it over zirconium chips at 1000 F.

Many capsule experiments have been successfully completed. A summary of these experiments appears in Table 1. Corrosion was evaluated by specimen weight change and microhardness tests. Both the capsule and specimens were examined microstructurally to ascertain the mechanism of corrosion.

The microhardness measurements on welded specimens are difficult to interpret because of the variation of hardness across the specimen and, therefore, have been omitted from the table. Results of the more recent experiments are also omitted because they have not been evaluated for microhardness. A trend, evident in the data presented is that columbium specimens sealed in nickel alloy capsules generally gained hardness after the test. Those sealed in columbium/1-percent zirconium alloy capsules generally softened, a condition which might be expected of annealed specimens.

TABLE 1

CAPSULE TEST RESULTS

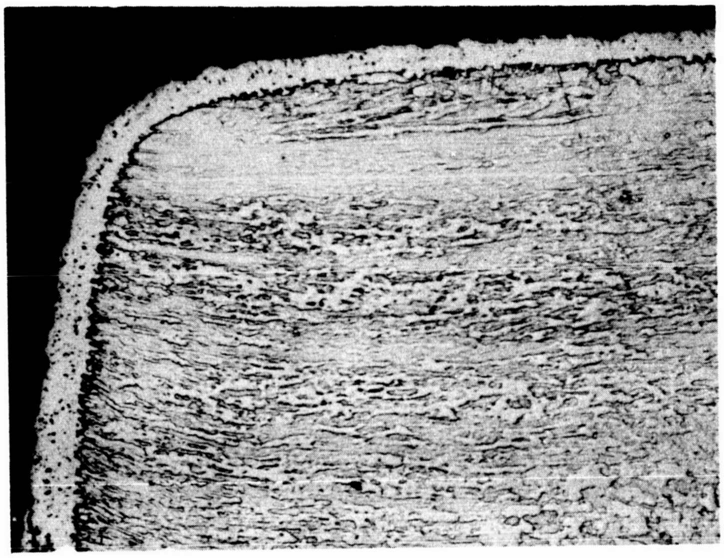
Temperature, F	Test Conditions: Time, hr	Test Conditions: Type of Test	Materials: Tab	Materials: Capsule	Tab Center Hardness KHN	Tab Edge Hardness KHN	Results: Tab Weight-Change Rate mg/cm²/day	Results: Metallographic Observations on Tab	Comments
As-Received			Cb		104	104			
As-Received			Cb/1%Zr		116	116			
1450	356	Rotating	Cb; weld	Hastelloy X			-0.44	no reaction layer	
1450	356	Rotating	Cb/1%Zr, weld	Hastelloy X			-0.39	no reaction layer	
1450	356	Rotating	Cb; weld	Inconel X			-2.32	no reaction layer	K contamination noted
1450	356	Rotating	Cb/1%Zr; weld	Inconel X			-1.90	no reaction layer	K contamination noted
1800	63	Static	Cb; weld	Hastelloy X			-1.88		K contamination suspected
1800	63	Static	Cb/1%Zr; weld	Hastelloy X			-2.89		K contamination suspected
1800	63	Static	Cb/1%Zr;wrought	Hastelloy X			-1.13		K contamination suspected
1800	100	Static	Cb; wrought	Hastelloy X	144	154	-0.32	0.3 mil reaction layer	
1800	100	Static	Cb; weld	Hastelloy X			-0.78	0.3 mil reaction layer 0 to 3 mil penetration	
1800	348	Static	Cb; wrought	Hastelloy X	200	200	-0.26	1 mil reaction layer	
1800	348	Static	Cb; weld	Hastelloy X			-0.29	1 mil reaction layer 0 to 5 mil penetration	
1900	50	Static	Cb; wrought	Hastelloy X	139	152	-2.80	0.5 to 1 mil reaction layer	K contamination suspected
1900	50	Static	Cb/1%Zr;wrought	Hastelloy X	116	143	-1.29	0.5 to 1 mil reaction layer	K contamination suspected
1900	63	Static	Cb; wrought	Hastelloy X			-7.23	1.5 mil reaction layer	K contamination suspected
1900	63	Static	Cb; weld	Hastelloy X	116		-5.30		K contamination suspected
1900	63	Static	Cb/1%Zr; weld	Hastelloy X			-2.90	3-5 mil reaction layer	K contamination suspected
								5-8 mil penetration	K contamination suspected
1900	159	Static	Cb; weld	Hastelloy X			-3.58		K contamination suspected
1900	159	Static	Cb/1%Zr; weld	Hastelloy X			-2.89		K contamination suspected
2000	24	Static	Cb; wrought	Hastelloy X	116	195	-0.83	no reaction layer	
2000	24	Static	Cb; weld	Hastelloy X			-1.43	no reaction layer	
2000	90	Static	Cb; wrought	Cb/1%Zr	87	70	specimen nicked	no reaction layer sharp corners	
2000	90	Static	Cb/1%Zr; wrought	Cb/1%Zr	98	123	specimen nicked	no reaction layer sharp corners	
2000	300	Static	Cb; wrought	Cb/1%Zr	83	92	-0.03	no reaction layer sharp corners	
2000	300	Static	Cb/1%Zr; wrought	Cb/1%Zr	83	77	+0.06	no reaction layer sharp corners	

From the weight-change measurements, the following conclusions can be drawn:

1. There is definitely a reaction between columbium and nickel-base super alloys when coupled by potassium above 1450 F.
2. In spite of the scatter in weight-change data, columbium/1-percent zirconium specimens were not attacked as rapidly as wrought columbium samples when coupled to nickel alloys. The only exception is the result from a 63-hour, 1800 F test; it cannot be explained at the present time.
3. There are definite indications that columbium specimens in columbium/1-percent zirconium capsules are not corroded by potassium (less than $\pm$ 0.05 mg/cm^2/day).

Figure 1 is a typical photomicrograph of the reaction layer observed on a columbium specimen coupled to a nickel alloy capsule by potassium. The layer has been sampled for X-ray diffraction identification, but the results are inconclusive because the diffraction patterns of the nickel/columbium intermetallic compounds have not been published. X-ray fluorescence identification is being attempted. The absence of a reaction layer at 2000 F (see Table 1) is not positively understood at the present time. Inasmuch as the layer does exist after tests at 1800 and 1900 F, it is thought that it flakes off at the higher temperature.

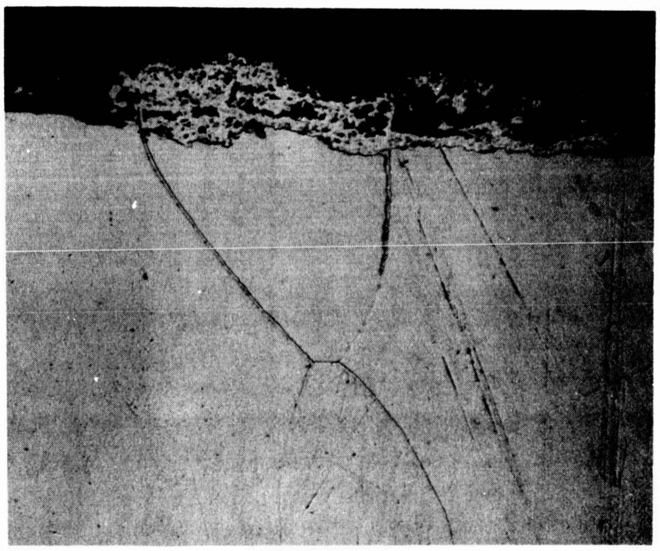
Intergranular attack has been found in several capsules where a columbium weld region contacts liquid potassium. A structure typical of this type of corrosion is shown in Fig. 2. This has been observed in columbium samples contained in both nickel alloy capsules and columbium alloy capsules. This type of corrosion was found only in weld regions. Several



250 X

Figure 1. Section Through Corner of Powder Metallurgy Columbium Corrosion Tab

The tab was tested for 63 hours at 1900 F in a potassium-filled, Hastelloy-X capsule. The etchant was 170 HNO_3, 50 HF, 5 citric acid, 510 methanol.



250 X

Figure 2. Section at Weld Bead of Arc-Melted Columbium/1-percent Zirconium Corrosion Tab

This tab was tested for 63 hours at 1900 F in potassium-filled, Hastelloy-X capsule. The etchant was 170 HNO_3, 50 HF, 5 citric acid, 510 methanol specimens have shown that the intergranular attack proceeded up to a fine-grained section and stopped. These observations delineate an important area for study. More experiments will be done to evaluate this phenomenon further.

The role of oxygen in the experimental results reported in Table 1 is not clear, but it may account for some of the scatter in the weight-change measurements. The correlation of data in one series of experiments and the lack of correlation between series may very well be due to variations in oxygen dissolved in the alkali metal introduced during capsule preparation. For example, a series run at 1900 F for 63 hours, which was allowed to stand over a weekend exposed to the dry box atmosphere, showed greater weight change than a series at 1900 F for 50 hours which sealed as promptly as possible. The dry box atmosphere was not perfectly pure, and probably introduced nitrogen or oxygen into the potassium before the capsule was sealed. Attempts will be made to ascertain the purity of the potassium before and after some of the future experiments.

A number of specimens of new columbium and molybdenum alloys have been received. These will be tested in columbium/1-percent zirconium capsules at temperatures around 2000 F in potassium.

An apparatus for dynamic capsule tests at 2200 F was described in the last quarterly progress report. This has been built and has been operating successfully for over 65 hours. However, there are no results to report at the present time.

LOW-TEMPERATURE LOOP STUDIES

The conversion of the sodium loop to operation with potassium was described in detail in the first quarterly progress report. These modifications have been completed.

Recently, the loop was operated successfully after a number of attempts to start the loop had failed. The starting procedure was to pressurize the sump and fill the loop with potassium. Potassium was circulated without boiling to clean all surfaces of the loop. Then the boiler was heated and circulation of boiling potassium initiated.

Difficulties were encountered during the circulation of liquid potassium. Circulation was interrupted by plugs formed at orifices or constrictions in tubing. Therefore, the loop could not be operated under boiling conditions. Soon after the first difficulties, it was discovered that the potassium had been received in a leaky container. Moreover, carbon deposits were found on heated lines from the shipping container, indicating the presence of an organic material. To rid the potassium of these impurities, the loop design was further modified by the addition of freeze traps and filters. A flow diagram of the loop (Fig. 3) shows its configuration as it is at this date. A small loop has been attached with a filter and hot zirconium chip trap for purification of the potassium prior to circulation in the main loop.

The low-temperature loop was recently operated successfully for 50 hours under boiling conditions with the boiler at 1800 F. Samples of molybdenum, wrought columbium, columbium/1-percent zirconium, and tantalum were suspended in the sample pot at the exit of the boiler. Samples of columbium

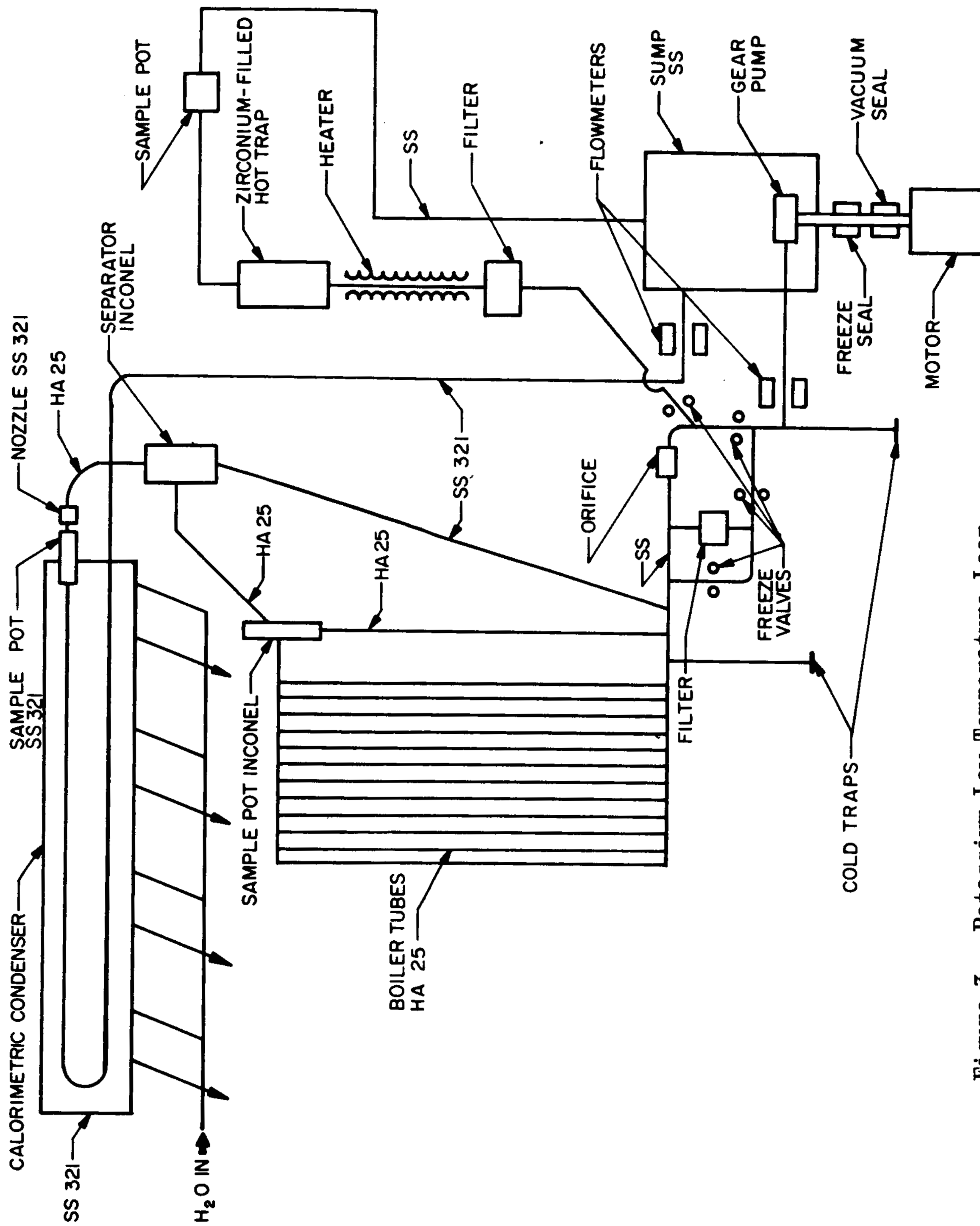


Figure 3. Potassium Low-Temperature Loop Schematic as of 11/22/60

and columbium/1-percent zirconium were suspended at the exit of the load nozzle. The velocity of the liquid through the nozzle prior to boiling operation was estimated to be approximately 100 ft/sec and, the subsequent vapor velocity may have been approximately Mach 2.5. The boiler temperature was approximately 1800 F and the condenser 1350 F.

The results for the above run are given in Table 2. Examination of the boiler specimens indicated that molybdenum, columbium/1-percent zirconium, columbium, and tantalum show levels of resistance to corrosion decreasing in that order. These results are not unexpected, since the sample pots in the loop are made of Inconel, a nickel alloy, and it is very likely that nickel transferred to the columbium specimen during the liquid circulation procedure. This transfer of transitional metals may have caused corrosion of the other specimens also.

Two samples placed in the sample pot downstream from the nozzle were not found after the test. Both were columbium samples. The exact cause has not been established. The sample pot itself did not show signs of corrosion; however, the test samples were gone. There is a possibility that the samples were destroyed by vibration in the high-velocity vapors or liquid.

The experiment described is being repeated with periodic checks on the samples by field radiography. Also a side loop has been provided for purifying the potassium by circulating it over hot zirconium prior to filling the main loop, thus avoiding high-velocity liquid circulation in the main loop.

Several potassium samples from the very first attempt to start the loop were submitted for chemical analysis. The samples were a collection of scrapings from the area around a plugged orifice. The results of the

TABLE 2

LOW-TEMPERATURE BOILING POTASSIUM LOOP RESULTS

Boiler Samples

	Approximate Temperature, F	Approximate Exposure Time, hours	Weight-Change Rate, $mg/cm^2/day$
Wrought Columbium	1800	50	-77.997
Cb/1%Zr	1800	50	-66.257
Molybdenum	1800	50	-7.755
Tantalum	(missing)		

Nozzle Samples

Both specimens were lost during the test.

analysis are shown in Table 3. Part of the sample that was soluble in HCl was analyzed by wet chemical methods. Acid insolubles were collected and analyzed by spectroscopic methods. The total estimate is based on the sum of both results. The probable sources of the major impurities are indicated in the last column. The only constituent having an unknown origin is silver. Some of the contituents may have been in the form of solids in the liquid potassium and, therefore, do not represent true solubilities.

HIGH-TEMPERATURE COLUMBIUM LOOPS

Two loops are to be fabricated of columbium/1-percent zirconium alloy to study the compatibility of materials at 2000 F boiler temperatures. The objective of this part of the program is to establish the applicability of the columbium alloy as a suitable material for use in high-temperature, potassium, space power plants. Since the results of the capsule tests have proven that the mixture of super alloys with columbium may lead to accelerated corrosion of the columbium/1-percent zirconium loop walls, a major change in the initial design criteria was necessary.

Potassium Pumps

As indicated in the first quarterly report, several types of pumps were being considered. The gear pump was favored at first because of the successful experience with this type of pump on the low-temperature loop. Also, the pump would be relatively inexpensive if made from transition metal super alloys. The pumps in these two loops will be operating at 1350 F and 1500 F. Although no layer was detected on columbium specimens

TABLE 3

CHEMICAL ANALYSIS OF POTASSIUM SCRAPINGS
AROUND PLUGGED ORIFICE

Element	6NHCl Soluble ppm wt	HCl Insoluble*	Total Concentration ppm	Probable Source
AG	500	M	> 500	Not known
Al	300,50	T	50	
B	300,10	S	10	Glass
Si	5000,50	---	50	
Cu	1000 - 2500	S	1000 - 2500	Drying Agent
Cb	800	S	800	
Zr	300,100	S	200	
Ni	< 50	T	< 50	
Cr	100	S	100	Loop Materials and Specimens
Fe	50	S	50	
Co	< 50	T	< 50	
Mo	50	T	50	
Be	< 1	T		
Cd	50	S		
Cu	30	T		
Mg & Mn	5	T		
Pb	50	T		
Su	< 100	NR		
Ti	50	NR		
V	< 100	NR		
Li & Zn	< 100	---		

*M = Major Constituent, S = Minor Constituent, T = Trace, NR = No Reading sealed in nickel alloy capsules at 1450 F, there was a detectable weight loss. Moreover, any nickel dissolved in the pump cell would react with columbium in the boiler and possibly accelerate corrosion there. Other transition metals may have a similar affect, but there is not sufficient time to explore all possibilities. Therefore, the search for a pump design not introducing transition metals was intensified.

An immediate suggestion was to fabricate the gear pump from columbium or other refractory alloys. However, this would be very expensive and none of the alloys has been proven for this application. Therefore, this solution did not appear promising.

Different types of centrifugal pumps were considered. The venturi pump and the turbopump had the right characteristics for the application (300 psi at 0.2 gpm). These reduced the requirements on columbium alloys but increased the problem of seals because of their shaft velocities. This also did not quite answer the need.

Electromagnetic pumps were reconsidered. At first, the linear pump was the type that suggested itself. However, a brief review of its characteristics revealed that this pump could not create the pressure head required unless multiple series units were used. Moreover, the attachment of conductors directly to the columbium, and the small gap requirements, made it impossible for this application.

Recently, the electrodynamic-type pump was suggested and investigated. In this pump design, the pump cell is a loop of tubing. The tubing is placed between electromagnets that are rotated by a motor. The magnetic field that passes through the tubular pump cell provides magnetic vanes to cause the liquid metal to move. This type of pump can supply the high-head/low-flow characteristics demanded in this application. The pump cell presents no seal problems and, moreover, does not introduce foreign metals into the loop. Pumps of this type have operated at 1500 F successfully. Therefore, this pump has been selected for the high-temperature columbium loops.

Design of Boiler

After considering several heating methods, a conduction method was chosen. A schematic drawing of the boiler is shown in Fig. 4. The boiler wall is made from 2-inch OD, 1/4-inch wall, columbium/1-percent zirconium tubing and heated by special calrod-type heaters immersed in a sodium-filled jacket (to improve thermal conductivity). The heaters consist of sheaths of columbium/1-percent zirconium and heating elements of molybdenum embedded in MgO insulation. Surrounding the boiler is a clam-shell heater to supply the heat lost by radiation. Further insulation is provided by tantalum radiation shields. Several thermocouples will be located in the thermocouple wells located above and below the liquid interface. Specimens are hung from the thermocouple well projecting through the top of the boiler. The thermocouple well also supports baffles for removing entrained liquid in the vapors. The potassium inlet was purposely located on the side to provide a trap for solid precipitates in the boiler.

Design of Condenser

The design of the condenser is similar in general construction to that of the boiler. Stainless steel water tubes are immersed in a lead-filled jacket. This surrounds a 1-inch-diameter columbium/1-percent zirconium tube. Thermocouple wells are located as in the boiler, and similarly are used to suspend specimens in the liquid.

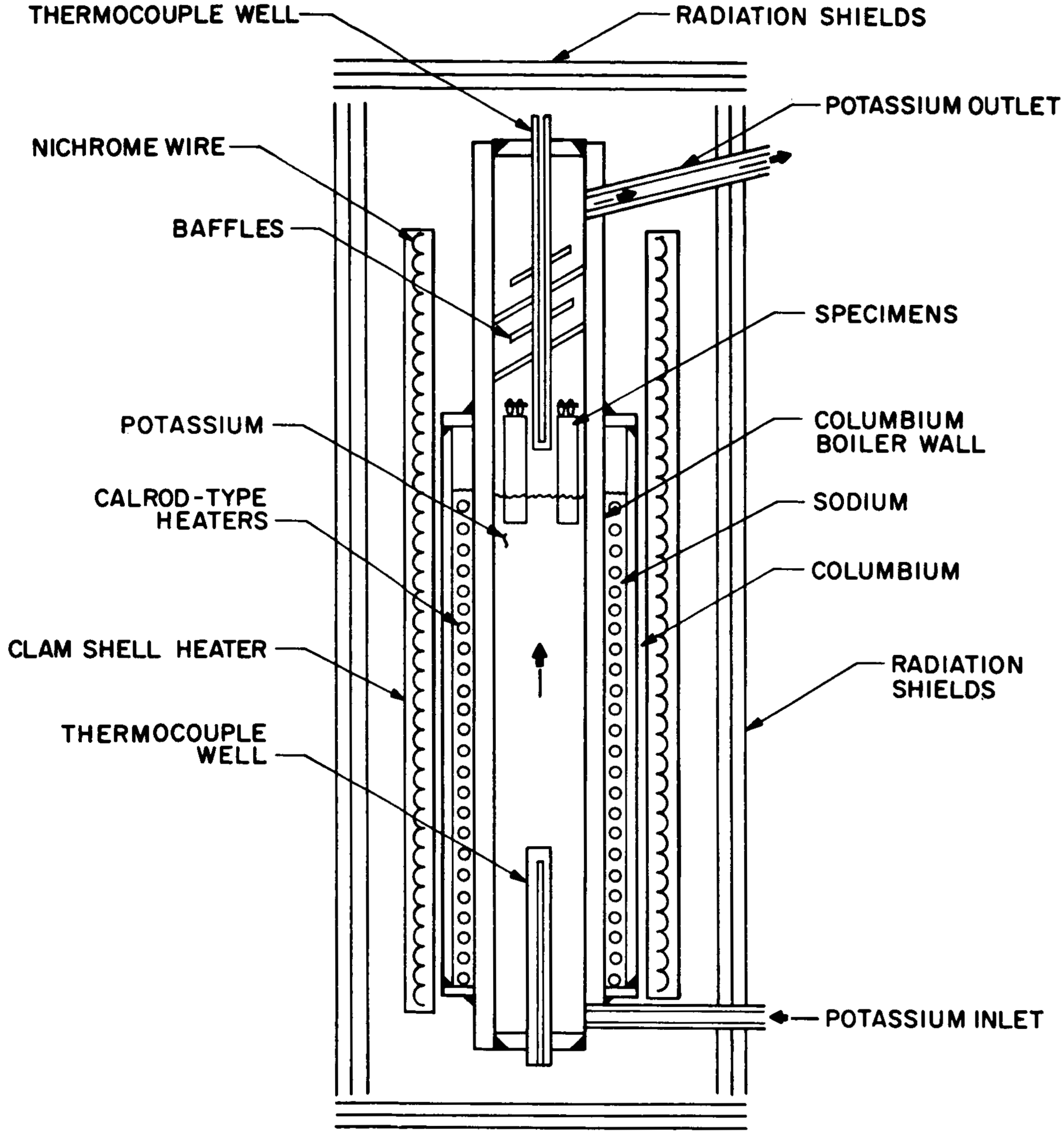


Figure 4. Colubium Boiler Design

Over-All Design

Figure 5 shows the over-all arrangement of the loop in the vacuum vessel. The boiler and condenser are joined at the top through a nozzle designed to produce the proper back pressure at the desired volume throughput. More specimens will be inserted in the tubes on both sides of the nozzles. Constrictions in the boiler feed line and the condenser return lines will provide the linear flows of 1 ft/sec and 8 ft/sec, respectively. A magnetic flowmeter will be placed on the boiler feed line.

Control of Loops

The temperature of the boiler will be controlled by means of a thermocouple located in the boiler. A boiler liquid level gage will regulate the coil current in the magnetic pump, thus changing the velocity of liquid potassium on demand.

GENERAL PLAN FOR CONSTRUCTION AND OPERATION OF LOOPS

The procedures for assembly and operation are being formalized, and will be presented in more detail in the next quarterly progress report. The procedures will be as outlined below:

Construction

The loop will be essentially a sealed capsule. Potassium will be sealed under vacuum.

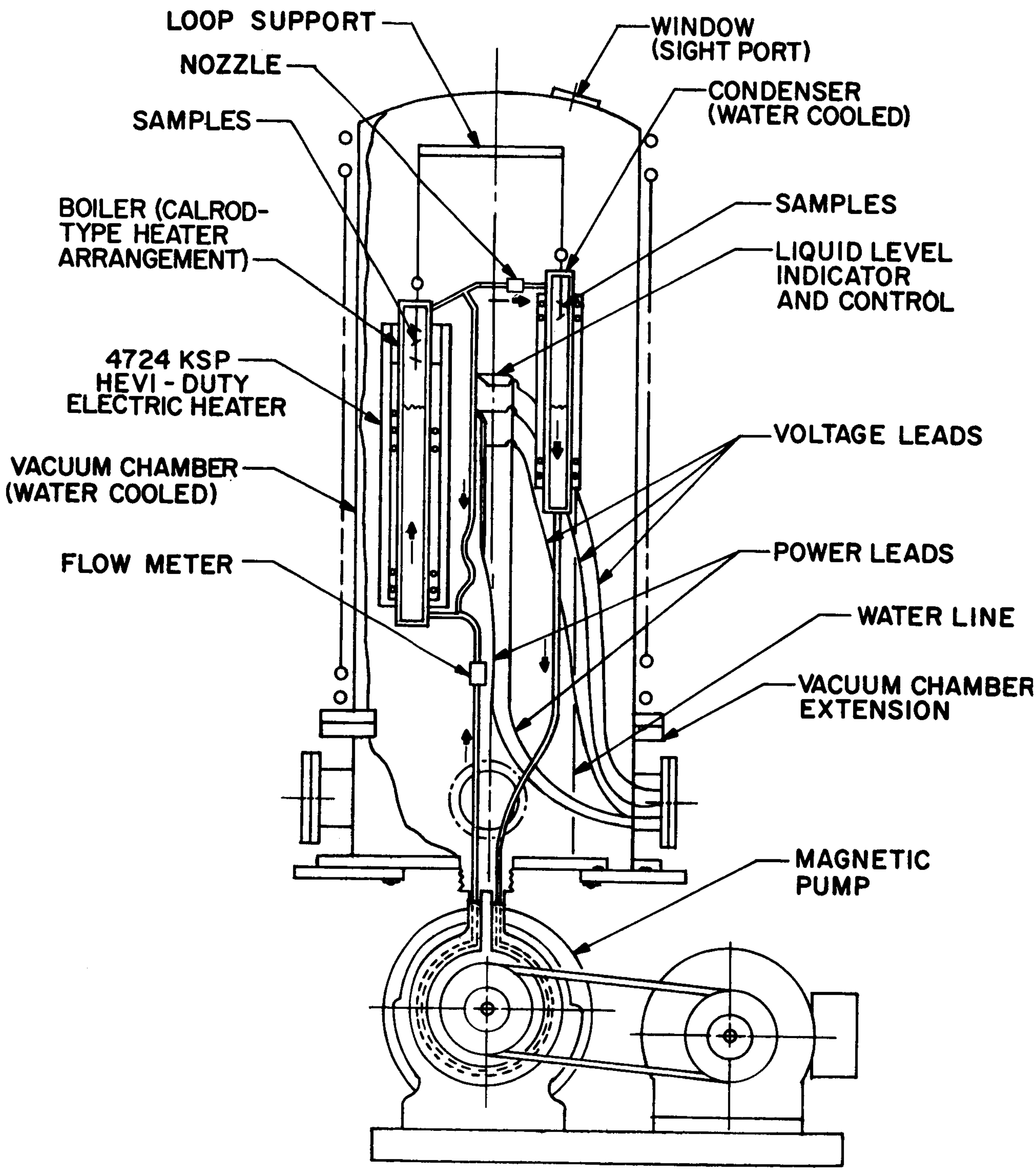


Figure 5. Cb-K Loop (Using Magnetic Pump)

All welding will be done under a standard, purified argon atmosphere.

All components will be leak checked prior to being incorporated in the loop. Helium leak detectors will be used.

Flowmeters, liquid level gauges, and pumps will be tested prior to loop assembly.

Specimen selection will be limited to the basic refractory metals: columbium, molybdenum, tungsten, and tantalum.

After the loop and associated parts have been placed in the vacuum chamber, the ambient temperature of the system will be raised to melt all potassium in the loop from the top down. The loop will be outgassed under vacuum at temperatures up to 1000 F, after which the vacuum chamber will be isolated under vacuum.

Operation

The loop will be cooled periodically and examined by gamma radiography.

The test specimens will remain in the loop for the entire life of the loop.

The loop will be controlled in the same way the current low-temperature loop is controlled. A boiler level device will control the electromagnetic pump and the boiler temperature will be regulated.

The loop will be disassembled and examined thoroughly after completion of the operation.

It is estimated that the total test period of the loop will be about 500 hours under full operating conditions, unless the radiographic inspection indicates the need for earlier termination of the test run.

WELDING STUDIES

Since the loops will be of all welded construction, a small program was initiated to check welding procedures to be used. A small inert-atmosphere box to be used for loop fabrication has been completed. The argon used in the box is purified by bubbling the gas through NaK. Samples of columbium/1-percent zirconium alloy tubing and sheet were welded in the box and examined microstructurally and by tensile test. Microstructurally, the welds were clear with only a few particles visible at high magnifications. A similar weld, made intentionally in air, showed a network of particles in a subgrain structure. In both weld specimens, the heat-affected zone was also clear of particles of second phase while the parent metal exhibited a fine dispersion of particles.

Tensile test specimens were prepared from a longitudinal section of welded 1-inch-diameter columbium/1-percent zirconium tubing and were compared with a similar specimen of the parent metal. One specimen of the weld was ground flat. The three specimens are shown in Fig. 6 as they appeared after the test. Both of the weld specimens broke in the heat-affected zone. However, the flattened specimens showed signs of ductility in the weld itself. The results of the tests are given in Table 4. They show that the heat-affected zone had one-half the strength and twice the elongation of the parent metal. The strength of the heat-affected zone is practically the same as that of an unalloyed columbium. The intergranular attack observed in weld areas after the capsule tests is cause for even greater concern.

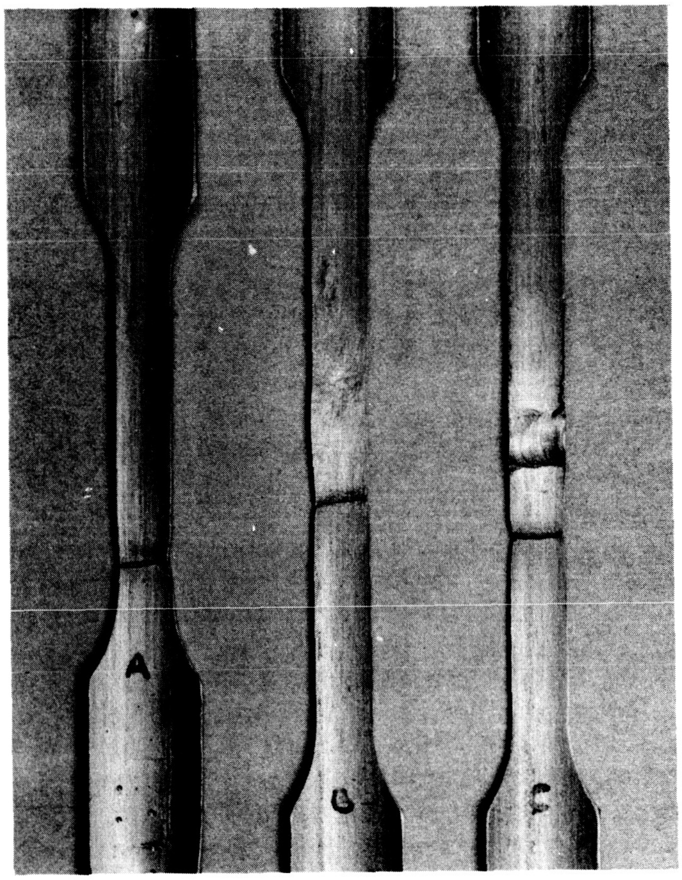


Figure 6. Longitudinal Weld Tensile Specimens

A. Parent metal

B. Weld ground flat

C. Welded tubing

TABLE 4

RESULTS OF TENSILE TESTS ON WELDED COLUMBIUM/ZIRCONIUM

Specimen	Yield Strength psi x 1000	Ultimate Strength, psi x 1000	Elongation, percent
A	35.6	51.4	14.0
B	35.8	49.6	15.0
C	75.8	90.0	8.0

Some heat treatments of the welds will be attempted to promote precipitation, with the hope that the strength and corrosion resistance of the weld region will be improved.

FLOW GAGE CALIBRATION

Equipment is being built to test the magnetic flow gage. Because of the vacuum surrounding the columbium/1-percent zirconium tubing, a permanent magnet flowmeter, similar to those used on the boiling sodium and low-temperature potassium loops, would create the least outgassing problem. The signal from these gages is low, particularly at low flowrates. Columbium also introduces an additional problem by its low electrical resistance. Therefore, tests are necessary to establish the accuracy of the meter.

PURIFICATION OF POTASSIUM

Since no provisions for purification will be provided in the loop, all potassium used will have to be purified first. A procedure for this operation is being formalized.

THERMOCOUPLE EVALUATION TESTS

Apparatus for testing thermocouples was built some time ago, but the activities were delayed until recently. Experiments are being conducted, and results will be forthcoming in the next quarterly progress report.

CURRENT STATUS AND FUTURE ACTIVITIES

The program has been reviewed and a new schedule (Fig. 7) devised for the construction of the columbium loops.

The capsule test evaluation program at Atomics International will be continued through to mid March when all the effort will be diverted to analysis of samples from the columbium loops. The program during this period will have the following objectives:

1. Studies on the intergranular corrosion of columbium and its alloys in welded regions.
2. The role of oxygen dissolved in potassium.
3. Capsule determinations of the solubility of columbium and other refractory metals in potassium.

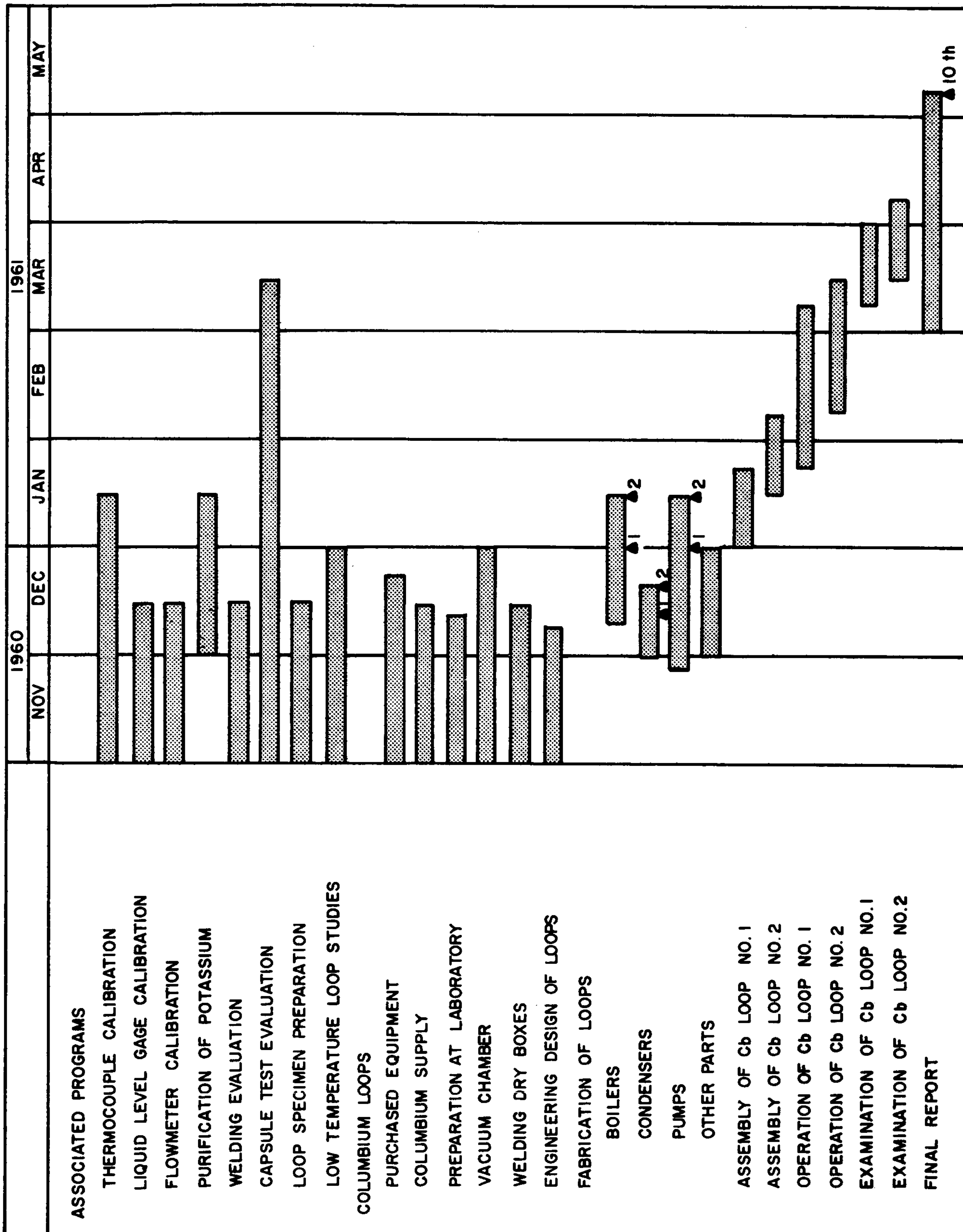


Figure 7. Revised Schedule for Boiling Potassium Loops

4. High-temperature burst tests.
5. Dynamic corrosion tests at 2200 F.
6. Analysis of specimens from the low-temperature loop

Specimens for the columbium loops will be ordered and prepared for insertion in the loops by the middle of December 1960.

It is planned that the low-temperature loop studies will be terminated by the end of December. The last run will be used to evaluate refractory metals in contact with potassium vapor (with the minimum pretest contact with flowing liquid potassium) since transfer of nickel has been detected in capsule tests.

The high-temperature columbium loops development will be moving from the design stage into the fabrication stage. All of the materials needed preliminary to installing the first columbium loop will be received by the end of December 1960. The purchased equipment includes such items as the recorders and controllers, leadthroughs, heaters, calrod-type heaters for the boiler, and thermocouples.

An order for 2-inch-diameter, 1/16-inch-wall tubing has been cancelled because of fabrication difficulties at Wah Chang, and the loop has been redesigned to incorporate "off the shelf" forms which will be delivered by mid December.

The work of installing water lines, power supply, and laboratory equipment, including instrumentation panels, will be complete by the middle of December. One vacuum bell jar is complete, and the revisions for the first loop and the vacuum chamber for the second loop have been designed. These are scheduled for completion by the end of December.

Two welding dry boxes are needed to complete the construction of the loops. A small one, now complete and in operation, has been providing satisfactory welds. The boilers and condensers for the two loops will be fabricated in this box. Assembly of the entire loop will be accomplished in a large dry box which will be completed around the middle of December.

The design of the loop has been frozen, and detailed engineering drawings are being prepared. These will be finished early in December. Fabrication of the boiler, condensers, pumps, and other parts are scheduled (Fig. 7). The first pump has been ordered, and the second order will be placed early in December.

The first columbium loop will be assembled, filled with potassium and installed in the vacuum chamber by the latter part of January 1961. The second loop will reach that stage early in February.

The remainder of the contract period will be utilized to operate the loops, examine the dissected loop, and report results of the loop experiments and capsule studies.